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CONDUCTIVE RESIN SHEET FOR ELECTRODE OF BATTERY AND ITS METHOD OF  
MANUFACTURE  
[Denchi denkyoku yo dodensei jushi shiito oyobi sono seizo soso]

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[Claim 1] A conductive resin sheet comprising a thermoplastic resin containing conductive particles; said conductive resin sheet for an electrode of a battery characterized by the volume resistivity of said conductive resin sheet being 100  $\Omega\cdot\text{cm}$  or less, the water vapor permeability being less than  $50 \text{ g}/(\text{m}^2\cdot\text{day})$ , and comprised by depositing an electrode active substance on a least one side thereof.

[Claim 2] The conductive resin sheet for an electrode of a battery of Claim 1 characterized by containing 10% by weight or more of at least one kind of conductive particle selected from among carbon, metal and a metal compound as the conductive particle.

[Claim 3] The conductive resin sheet for an electrode of a battery of Claim 2 characterized by the metal compound being tin oxide and/or indium oxide.

[Claim 4] The conductive resin sheet for an electrode of a battery of Claim 2 characterized by the metal oxide being at least one kind of metal compound selected from any of a carbide, nitride, boride and silicide of at least one kind of metal chosen from Ti, Zr, V, Nb, Ta, Cr, Mo and W.

[Claim 5] The conductive resin sheet for an electrode of a battery of any of Claims 1 to 4 characterized by the conductive particles having a core/shell structure.

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\* Claim and paragraph numbers correspond to those in the foreign text.

[Claim 6] The conductive resin sheet for an electrode of a battery of any of Claims 1 to 5 characterized by the conductive particles being needle shaped and the particle major axis/particle minor axis ratio being a range of 5 to 50.

[Claim 7] The conductive resin sheet for an electrode of a battery of any of Claims 1 to 6 characterized by the electrode active substance being lead metal and/or lead oxide.

[Claim 8] The conductive resin sheet for an electrode of a battery of any of Claims 1 to 7 characterized by the resin composing the conductive resin sheet being at least one kind of resin selected from among polyethylene, polypropylene, polystyrene, and copolymers of these.

[Claim 9] The conductive resin sheet for an electrode of a battery of any of Claims 1 to 8 characterized by having at least two layers wherein the types of conductive particles contained are different.

[Claim 10] The conductive resin sheet for an electrode of a battery of any of Claims 1 to 9 characterized by depositing respectively different electrode active substances on the surface of the conductive resin sheet.

[Claim 11] A method for manufacturing a conductive resin sheet for an electrode of a battery characterized by comprising at least the steps of: obtaining a conductive resin sheet by extruding a thermoplastic resin composition containing conductive particles into

a sheet shape through a spinneret of an extruder, and cooling and solidifying it with a cooling medium; and crimping an electrode active substance to the surface of the resultant conductive resin sheet.

[Claim 12] The method for manufacturing a conductive resin sheet for an electrode of a battery of Claim 11 characterized by supplying a thermoplastic resin composition wherein the types of conductive particles respectively contained in two or more extruders are different, introducing respective melted resins into a lamination block or lamination spinneret after a melt extrusion, and performing two or more laminations.

[Claim 13] The method for manufacturing a conductive resin sheet for an electrode of a battery of Claim 11 or 12 characterized by obtaining the conductive resin sheet and subsequently reducing the thickness of this sheet by rolling.

[Detailed Specifications]

[0001] [Technical Field of the Invention]

The present invention relates to a conductive resin sheet for an electrode of a battery which is lightweight and outstanding in chemical resistance and electroconductivity, and a method of manufacture thereof, and in further detail, a conductive resin sheet for an electrode of a battery having superior quality which is ideal as an electrode material for a lead storage battery, and a method for

manufacturing the conductive resin sheet for an electrode of a battery thereof.

[0002] [Prior Art]

A metal foil or plate, or a metal mesh or grid is used for battery electrodes conventionally, and a lead grid is used with a lead storage battery, while aluminum foil or copper foil is used with a lithium ion battery. Meanwhile, a grid for an electrode whose weight was reduced to improve the weight energy density of the battery has been proposed in Tokukai JP-A No. H10-188996. The grid for this electrode is provided with a conductive portion comprising lead or a lead alloy as a mesh-like substance comprising glass fibers, etc.

[0003] [Problems to be Solved by the Invention]

However, with such a conventional battery electrode, drawbacks existed, as follows. That is, although a metal foil, plate, mesh or grid was conductive, the density of the metal was higher than that of a resin, and when a metal material was used as the electrode material for a battery, the electrical output per weight of the battery was low. Furthermore, with a lead storage battery, the electrode material comprising lead metal was corroded by repeated charging/discharging of the battery, which was an impediment to extending the battery life.

[0004] In addition, as in the case of a grid for an electrode proposed in Tokukai JP-A No. H10-188996, a metal material was required for the conductive member because the grid was composed of a

non-electroconducting member, and there was a problem because the electrode was still heavy.

[0005] An object of the present invention is to provide a conductive resin sheet for an electrode of a battery which eliminates the drawbacks of the above-mentioned prior art, is lightweight, can be used in a lead storage battery and is outstanding in chemical resistance and conductivity, and a method of manufacture thereof.

[0006] [Means for Solving the Problems]

As a result of meticulous investigations in view of the aforementioned problems, the inventors of the present invention discovered that the aforementioned problems could be solved by retaining the electrode active substances in the thermoplastic resin sheet containing conductive particles and having a specific volume resistivity and water vapor permeability, and they were led to achieving the present invention.

[0007] That is, the conductive resin sheet for an electrode of a battery of the present invention is a conductive resin sheet comprising a thermoplastic resin containing conductive particles; said conductive resin sheet for an electrode of a battery characterized by the volume resistivity of said conductive resin sheet being 100  $\Omega \cdot \text{cm}$  or less, the water vapor permeability being less than 50  $\text{g}/(\text{m}^2 \cdot \text{day})$ , and comprised by depositing an electrode active substance on a least one side thereof.

[0008] In addition, the method for manufacturing a conductive resin sheet for an electrode of a battery of the present invention a method for manufacturing a conductive resin sheet for an electrode of a battery characterized by comprising at least the steps of: obtaining a conductive resin sheet by extruding a thermoplastic resin composition containing conductive particles into a sheet shape through a spinneret of an extruder, and cooling and solidifying it with a cooling medium; and crimping an electrode active substance to the surface of the resultant conductive resin sheet.

[0009] [Embodiments of the Invention]

The preferred embodiments of the present invention will now be described.

[0010] The conductive resin sheet for an electrode of a battery in the present invention is such that the electrode active substance is deposited on at least one surface of a conductive resin sheet wherein the volume resistivity is 100  $\Omega \cdot \text{cm}$  or less and the water vapor permeability is less than 50  $\text{g}/(\text{m}^2 \cdot \text{day})$ .

[0011] In the present invention, a conductive resin sheet lower in weight than a metal material, which is a conventional current collector material, can be planned by composing the conductive resin sheet mainly of a thermoplastic resin, and by using this, the weight energy density of the battery can be improved.

[0012] The conductive resin sheet for an electrode of a battery of the present invention is composed of a conductive thermoplastic

resin sheet (conductive resin sheet) and an electrode active substance as such, but the water vapor permeability of the conductive resin sheet part must be less than  $50 \text{ g}/(\text{m}^2 \cdot \text{day})$ , and preferably at most  $40 \text{ g}/(\text{m}^2 \cdot \text{day})$ . When the water vapor permeability is greater than this, inconveniences develop when a battery is constructed in that evaporation of the electrolyte, leakage of the active substance, and so forth are likely to occur. The lower the value of the water vapor permeability, the more preferable it is. Although the water vapor permeability can be reduced by increasing the sheet thickness, the weight of the electrode also increases because the sheet thickness increases; hence, the lower limit of the value thereof is substantially  $1 \text{ g}/(\text{m}^2 \cdot \text{day})$ .

[0013] Furthermore, the volume resistivity of the thermoplastic resin sheet part of the conductive resin sheet for an electrode of a battery of the present invention must be  $100 \Omega \cdot \text{cm}$  or less. When the volume resistivity is greater than  $100 \Omega \cdot \text{cm}$ , the drop in the voltage of the battery is high, and the power loss is great. The smaller the volume resistivity, the more preferable it is. It is preferable that it be  $10 \Omega \cdot \text{cm}$  or less, and further,  $1 \Omega \cdot \text{cm}$  or less. In order to reduce the volume resistivity, increasing the content of the conductive particles is effective, but if the content is increased excessively, molding of the sheet itself becomes difficult, and defects, such as pinholes, are likely to occur. The limits of the volume resistivity are substantially about  $0.01 \Omega \cdot \text{cm}$ . In order to

achieve this kind of volume resistivity, it is preferable to set the content of the conductive particles with respect to the thermoplastic resin to 10% by weight or more. A range of 15 to 60% by weight is preferable from the standpoint of the conductivity and sheet moldability.

[0014] It is preferable use at least one kind of conductive particle selected from among carbon, metal and a metal compound for the conductive particles contained in the thermoplastic resin. Titanium, copper, lead, zinc, pewter, and so forth may be cited for the metal. Furthermore, at least one kind of metal compound selected from among tin oxide, indium oxide, and a carbide, nitride, boride and silicide of at least one kind of metal chosen from Ti, Zr, V, Nb, Ta, Cr, Mo and W can be used. In particular, tin oxide, TiC, TiN, TiB<sub>2</sub> or TiSi<sub>2</sub>, which are a carbide, nitride, boride or silicide of Ti, are preferable. These conductive particles may be used singly or by mixing them at any given proportion.

[0015] There are no particular limits to the particle diameter of the conductive particles, but a range of 0.01 to 10 µm is preferable from the standpoint of the dispersibility in the thermoplastic resin, and a range of 0.05 to 5 µm is more preferable.

[0016] The conductive particles may have a core/shell structure wherein there is conductivity on the surface of the particles, as a conductive substance covers the surface of the particles. When the specific gravity of the conductive particles is high, the specific

gravity of the conductive particles themselves can be reduced by making a core/shell structure, and a lightweight conductive resin sheet can be obtained even if the conductive particles are contained in a thermoplastic resin at a high percentage. Furthermore, if the shape of the conductive particles is needle shaped, a high conductivity is obtained with a smaller amount of particles. In this case, it is preferable that the particle major axis/particle minor axis ratio range from 5 to 50, and more preferably, 10 to 30. If the ratio is less than 5, the effects of the needle-shaped particles hardly materialize. Moreover, when it is greater than 50, the particles easily cohere in the thermoplastic resin. When it is difficult to make a needle-shaped structure with the conductive substance itself, needle-shaped conductive particles can be obtained by making a core/shell structure in which a conductive substance covers the needle-shaped particle surface, such as potassium titanate.

[0017] The thickness of the conductive resin sheet for an electrode of a battery of the present invention is not limited in particular, but handling during battery manufacture is difficult if the sheet is too thin. And if it is too thick, the weight per unit area of the sheet increases; hence, there are few effects for improving the weight energy density when it is used in the battery. Thus, the thickness of the conductive resin sheet part, excluding the electrode active substrate, preferable ranges from 20 to 1,000  $\mu\text{m}$ , and in particular, from 50 to 800  $\mu\text{m}$ .

[0018] The electrode active substance of the conductive resin sheet for an electrode of a battery of the present invention is deposited on at least one surface of the conductive thermoplastic resin sheet thereof.

[0019] The "electrode active substance" used in the present invention means a substance that participates in the chemical reaction in the battery. With a lead storage battery, lead oxide is used in the positive electrode, and moreover, lead metal is used in the negative electrode. A porous body comprising a powder is preferably used for the lead oxide.

[0020] The electrode active substance composing the conductive resin sheet for an electrode of a battery of the present invention is preferably lead metal and/or lead oxide. That is, it is preferable to use the conductive resin sheet for an electrode of a battery of the present invention in an electrode for a lead storage battery.

[0021] The amount of the electrode active substance deposited on the conductive thermoplastic resin sheet varies depending on the capacity required of a battery and is not limited in particular. But it is preferable that the thickness of the electrode sheet to which the active substance was deposited fall in a range of 0.1 to 5 millimeters, and more preferably, 0.3 to 3 millimeters from the standpoint of the handleability of the electrode sheet and reducing the weight of the battery.

For the method for depositing the electrode active substance on the conductive resin sheet, the electrode active substance molded into a sheet shape can be deposited by crimping it to the conductive resin sheet with a press or the like, and this may be heated even if crimping is performed at normal temperature. The conductive resin sheet of the present invention may be composed mainly of the thermoplastic resin; hence, the electrode active substance can be thermocompression bonded in the vicinity of the softening point of the thermoplastic resin. If the electrode active substance is, e.g., a lead sheet, it can be thermocompression bonded directly to the conductive resin sheet. Moreover, if the electrode active substance is lead oxide, a lead oxide paste is prepared by mixing and kneading dilute sulfuric acid in advance, this is made into a sheet, which is then dried to prepare a lead oxide active substance plate, which should be crimped to the conductive resin sheet.

[0022] In the present invention, there are no particular limits to the thermoplastic resin composing the conductive resin sheet. For example, polyethylene, polypropylene, polymethyl pentane, polystyrene, polyphenylene sulfide, polyvinyl chloride, polyethylene terephthalate, and the like may be used. But from the standpoint of chemical resistance, a resin comprising polyethylene, polypropylene, polystyrene, or a copolymer of these is preferably used in particular.

[0023] In the present invention, the conductive resin sheet may be constructed of at least two layers containing different types of

conductive particles. With such a configuration, in particular the layer containing particles outstanding in chemical resistance is used for the active substance, e.g., the electrolyte side, and a sheet in which chemical resistance and conductivity coexist at a high level can be obtained by using a layer containing particles outstanding in conductivity, but with poor chemical resistance, on the current collection side. In this case, there are no particular limits to the ratio of the thickness of each layer, and the layers may be laminated at any given ratio.

[0024] The electrode active substance should be deposited on at least one side of the conductive resin sheet of the conductive resin sheet for an electrode of a battery when a single electrode is constructed, but when a unit battery cell is constructed in a form connected in series, different types of electrode active substances for positive electrode use and negative electrode use may be deposited respectively on the front and back of the conductive resin sheet.

[0025] The method for manufacturing the conductive resin sheet for an electrode of a battery of the present invention is composed of at least two steps, with one being a step of obtaining the conductive resin sheet by extruding the thermoplastic resin composition containing conductive particles into a sheet shape through a spinneret of an extruder and cooling and solidifying this with a cooling medium, and the second step is a step of crimping the

electrode active substance to the surface of the resultant conductive resin sheet. In the first step, the cooling medium is preferably a metal drum or metal roll. It is preferable to adopt a structure in which a fluid for cooling flows inside these media. The temperature of the surface of the drum or roll can be adjusted by adjusting the temperature of the fluid for cooling. In order to obtain a conductive resin sheet outstanding in smoothness, it is necessary to bond a melted resin sheet extruded with these cooling media, but a conventionally-known method, such as an air knife, air chamber, nip roll or electrostatic impression method, may be used therefor.

[0026] When a polyolefin resin is used as the thermoplastic resin, an air knife system or air chamber system is preferably used. Furthermore, when the conductive resin sheet part of the conductive resin sheet for an electrode of a battery is composed of two or more layers, the thermoplastic resin composition having two different kinds of conductive particles contained is supplied to two or more extruders, and the respective melted resins are introduced into a lamination block or lamination spinneret after the melt extrusion, and a conductive resin sheet can be manufactured in a method in which two or more layers are laminated.

[0027] Since the thermoplastic resin containing a high concentration of conductive particles is unsatisfactory in moldability, the conductive resin sheet having the desired thickness may not be obtained by melt extrusion alone. In this case, after

obtaining the conductive resin sheet, the thickness can be adjusted to that desired by rolling the conductive resin sheet while pressurizing it between rolls.

[0028] The thermoplastic resin composition containing conductive particles may be obtained by melt kneading a conductor, such as carbon black, with a resin, such as polyethylene. Other additives, such as a blocking agent, expander, stabilizer, antioxidant, thinner/thickener, particle dispersant, and other resins certainly may be added, and kneaded. A conventionally-known device, such as a biaxial kneader, may be used for the melt kneading.

[0029] The conductive resin sheet for an electrode of a battery obtained in this way has outstanding conductivity and chemical resistance, and is preferably used as an electrode for a battery. The weight energy density of the battery can be improved by using the conductive resin sheet for an electrode of a battery of the present invention in a lead storage battery. In addition, the conductive resin sheet for an electrode of a battery of the present invention can be hot bonded by applying heat. This conductive resin sheet for an electrode of a battery can be hot bonded to a battery cell container to construct an airtight battery.

#### [0030] [Method for Measuring Physical Properties]

The methods of measurement used in the present invention are described next.

[0031] 1. Volume resistivity of sheet

This was measured in a four-probe method in accordance with the JIS K-7194 method.

[0032] 2. Water vapor permeability

This was measured at conditions including  $25\pm0.5^{\circ}\text{C}$  and a relative humidity of  $90\pm2\%$  RH using a Permatran-W3/30 manufactured by Modern Controls, Inc. in accordance with the JIS K-7129 method.

[0033] 3. Particle shape

The major axis and minor axis of the particles were measured by observing the particles under a scanning electron microscope and a transmission-type microscope, and the average for 1,000 particles was found.

[0034] 4. Particle diameter

The diameter of a circle having a surface area equivalent to the cross-sectional area of a particle was found by observing the particles under a scanning electron microscope and a transmission-type microscope, and the average for 1,000 particles was found.

[0035] [Practical Examples]

The present invention will be described in further detail according to the practical examples.

[0036] [Practical Example 1]

65 parts by weight of a low-density polyethylene and 35 parts by weight carbon black particles having a particle diameter of  $0.02\ \mu\text{m}$  were mixed, and melt kneaded with a biaxial kneader to obtain a

conductive resin composition. This resin composition was supplied to an extruder having a 90 millimeters cylinder diameter, melt extruded at 250°C, and extruded onto a metal drum as a molten sheet through a 400 millimeter wide T-die. The diameter of the metal drum was 500 millimeters and the surface temperature was 60°C. The molten sheet was adhered to the metal drum with an air knife. A 100 µm thick conductive resin sheet having a volume resistivity of 5 Ω·cm and a water vapor permeability of 10 g/(m<sup>2</sup>·day) was obtained using the above process.

[0037] Next, a 9 cm square, 500 µm thick lead sheet lead sheet was superposed on one surface of the 10 cm square conductive resin sheet obtained previously, leaving 5 millimeters on the outside of the conductive resin sheet, and pressed at a pressure of 1 MPa and a temperature of 120°C to obtain a 600 µm thick electrode sheet for the negative electrode of a lead battery.

[0038] [Practical Example 2]

Except for changing the carbon black particles to tin dioxide particles having a 0.02 µm particle diameter, a 100 µm thick conductive resin sheet having a volume resistivity of 15 Ω·cm and a water vapor permeability of 12 g/(m<sup>2</sup>·day), was obtained as in Practical Example 1. Furthermore, 30 parts by weight of a 30% aqueous sulfuric acid solution and 70 parts by weight lead oxide were mixed and kneaded well, whereby a lead oxide paste was prepared, injected into a form and dried, whereby a 500 µm lead oxide plate was obtained.

A conductive resin sheet containing the 9 cm square lead oxide plate obtained by the above process and a 10 cm square tin dioxide was superpositioned thereon, and pressed at a temperature of 120°C and a pressure of 1 MPa to obtain a 60 µm electrode sheet for the positive electrode of a lead battery.

[0039] The resultant electrode sheet was more lightweight than an electrode made of metal. Next, upon using 30% concentrated sulfuric acid as the electrolyte, the sheet of Practical Example 1 as the negative electrode and the sheet of Practical Example 2 as the positive electrode, and hot bonding the outer peripheral portion of each electrode to construct an airtight lead storage battery, the weight energy density of the battery was improved. Furthermore, no corrosion of either electrode was observed after charging/discharging of the battery.

[0040] [Comparative Example 1]

A resin composition in which the compounded proportions in Practical Example 1 were changed to 65 parts by weight carbon black and 35 parts by weight low density polyethylene was obtained. The resin composition could not be melt extruded; hence, it was pressed at 5 MPa as it was heated to 280°C, and a 100 µm thick sheet was obtained. Although the volume resistivity of this sheet was 0.1 Ω·cm, the water vapor permeability was 100 g/(m<sup>2</sup>·day). A sheet for a battery electrode was obtained by crimping the tin dioxide sheet as in Practical Example 2. A battery was constructed by substituting the

positive electrode in Practical Example 2 with the sheet in Comparative Example 1, but leakage of electrolyte occurred.

[0041] [Practical Example 3]

Except for using the conductive resin composition in which the tin dioxide particles were changed to titanium disilicide ( $TiSi_2$ ) particles having a particle diameter of 2  $\mu m$ , a 600  $\mu m$  thick conductive resin sheet for an electrode of a battery having a 1  $\Omega \cdot cm$  volume resistivity and a 20  $g/(m^2 \cdot day)$  water vapor permeability and containing a 100  $\mu m$  thick conductive resin sheet and an active substance was obtained as in Practical Example 2. Furthermore, a lead storage battery was constructed by using this sheet as the positive electrode and the sheet of Practical Example 1 as the negative electrode. Furthermore, no corrosion of either electrode after charging/discharging of the battery was observed.

[0042] [Practical Example 4]

The conductive resin composition used in Practical Example 1 was extruded from a first extruder and the conductive resin composition used in Practical Example 3 was extruded from a second extruder, respectively, at 250°C, and laminated as two layers using a lamination block.

[0043] The laminated melted resin sheet was extruded onto a metal drum as a melted sheet from a 400 millimeter wide T-die. The metal drum had a 500 millimeter diameter and surface temperature of 60°C. The melted sheet was bonded to the metal drum with an air knife.

A 100  $\mu\text{m}$  thick conductive resin sheet having a  $3 \Omega\cdot\text{cm}$  and a  $15 \text{ g}/(\text{m}^2\cdot\text{day})$  water vapor permeability was obtained by the above process. This conductive resin sheet included a 75  $\mu\text{m}$  layer compounded with carbon (carbon layer) and a 25  $\mu\text{m}$  thick layer compounded with titanium disilicide (titanium disilicide layer). Next, a 500  $\mu\text{m}$  thick lead sheet was crimped to the carbon layer side, as in Practical Example 1, and a 500  $\mu\text{m}$  thick lead oxide sheet was crimped to the titanium disilicide layer, and a 1,100  $\mu\text{m}$  thick electrode sheet was obtained. The sheet of Practical Example 1 and the sheet of Practical Example 3 were combined to construct a lead storage battery in which two battery unit cells were joined in series. In addition, no corrosion of either electrode after charging/discharging of the battery was observed.

#### [0044] [Practical Example 5]

Except for containing 20% by weight particles coated with tin dioxide on the surface of the needle-shaped titanium oxide having a major axis of 3  $\mu\text{m}$ , where the particle major axis/minor axis ratio was 14, a sheet for a battery electrode having a volume resistivity of  $10 \Omega\cdot\text{cm}$  and a water permeability of  $15 \text{ g}/(\text{m}^2\cdot\text{day})$  (conductive resin sheet thickness: 100  $\mu\text{m}$ ; lead oxide sheet thickness: 500  $\mu\text{m}$ ) was obtained as in Practical Example 2. A lead battery was constructed by combining the sheet of Practical Example 5 used as the positive electrode and the sheet of Practical Example 1 used as the negative

electrode. In addition, no corrosion of either electrode after charging/discharging of the battery was observed.

[0045] [Advantages of the Invention]

According to the present invention, a lightweight battery electrode outstanding in chemical resistance and conductivity is obtained, and a more lightweight battery can be planned; hence, the weight energy density of a lead storage battery can be improved.